

WaterSentinel Online Water Quality Monitoring as an Indicator of Drinking Water Contamination

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### **Executive Summary**

WaterSentinel (WS) serves as a demonstration project, or pilot, for designing and implementing an effective contamination warning system (CWS) in a drinking water distribution system. As part of WS, a CWS should be developed that encompasses monitoring technologies and detection strategies, combined with enhanced public health surveillance to collect, integrate, analyze, and communicate information to provide a timely warning of potential water contamination incidents and initiate response actions to minimize public health and economic impacts. The success of a CWS, and hence WS, depends on the ability to effectively integrate these components and analyze the resulting information in a timely manner to inform response actions that can substantially reduce the potential consequences of a contamination incident

Current means of monitoring water quality parameters, such as periodic grab sampling for disinfectant residual and infrequent sampling and analysis for a small number of specific contaminants, may be of limited scope and usefulness in the WS-CWS. Therefore, the WS-CWS aims to detect contaminants by utilizing a network of online water quality sensors, deployed throughout a drinking water distribution system, that are responsive to many contaminants. In many cases, information from online water quality sensors should provide the first indication of possible contamination, and should set into motion response actions to either corroborate or rule out contamination.

Establishing which water quality parameters provide the broadest coverage and most reliable indication of contamination is of critical importance to the success of this component of the WS-CWS. Research, conducted by online water quality sensor manufacturers and the EPA provided information about the potential of various contaminants of concern to produce detectable changes in specific water quality parameters. While there are numerous parameters that respond to contamination, a literature review found that the most effective parameters for detecting the 33 WS Baseline contaminants are free chlorine, total organic carbon (TOC), conductivity and pH. Oxidation/reduction potential corroborates chlorine sensor results. Other parameters such as chloride, nitrate and ammonia have been observed to change in the presence of contaminants but mostly due to interference of concomitant ions. Turbidity, which can be highly variable, is not a good primary indicator of contamination.

Utilizing online water quality monitoring as an indicator of drinking water contamination should be an integral part of the WS system architecture. Online monitoring should enable water utilities to detect potential contamination quickly and launch an appropriate response. Future work on online monitoring of radionuclides, a broader range of chemical and biological agents, and the use of event detection software should improve detection as the WS-CWS evolves.

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### **Section 1.0: Introduction**

A successful contamination warning system (CWS) should include components that yield timely and reliable warning of a potential contamination incident. Drinking water utilities routinely monitor a number of water quality parameters (i.e., pH, conductivity, free chlorine, oxidation-reduction potential (ORP) and total organic carbon (TOC) in drinking water treatment and distribution. Under routine operations, however, these parameters are monitored only periodically through grab sampling programs, and at a frequency that would not provide early warning of a potential contamination incident. To employ conventional water quality monitors in a CWS, WaterSentinel (WS) proposes the application of multiple online water quality probes and sensors configured into sensor stations, placed throughout the distribution system, transmitting usable information in real-time. Questions that should be addressed for successful implementation of online water quality monitoring as a component of the WS pilot include:

- Do water quality parameters change in response to contaminants that could be introduced into the distribution system and, if so, which parameters are the best indicators of contamination?
- How can true contamination events be detected from the normal background variability of these parameters when deployed for extended periods of time?
- For a given distribution system, what are the optimal number of, and locations for, these sensor stations that would provide the best coverage and protection of the population served (sensor network design)?

The information provided in this document addresses the first question; do water quality parameters change in response to contaminants that could be intentionally or accidentally introduced into the distribution system and, if so, which parameters are the best indicators of contamination? The second and third questions relating to online water quality monitoring are addressed in *Overview of Event Detection Systems for WaterSentinel* (USEPA, 2005a) and *WaterSentinel System Architecture* (USEPA, 2005b), respectively.

This document describes the state-of-the-science of real-time, online water quality monitoring using conventional water quality parameters. The results from controlled studies have demonstrated that certain water quality parameters respond rapidly and predictably to contaminants of interest to water security, at concentrations well below the  $LD_{50}$  (a dose that results in death in 50% of the population exposed to that dose), and that, when configured to be used online, these online monitors promise to be an important component of the WS contamination warning system (WS-CWS).

The remaining sections of this document describe the following aspects of online water quality as an indicator of drinking water contamination:

- **Section 2.0: Background.** Presents an overview of manufacturers of online water quality monitors, the benefit of multiple sensors, and how various water quality parameters are measured.
- Section 3.0: Literature Review. Provides a description of research using online water quality monitors to detect contamination events using vendor and American Water Works Association Research Foundation (AwwaRF) studies as well as EPA studies.
- Section 4.0: Case Study. Describes the testing of two different modes of distribution pipe simulation in a pilot scale system.
- **Section 5.0: Conclusions.** Summarizes and concludes the use of online water quality as an indicator of drinking water contamination.

- **Section 6.0: References**. This section provides a bibliography of the references cited in this document.
- Appendix A: Acronyms.

A complete glossary of terms related to online water quality and the WS program is available in *WaterSentinel System Architecture* (USEPA, 2005a).

# Section 2.0: Background

Ideally, the monitoring devices used in a water distribution CWS would detect any and all of the possible agents that could be encountered. With the vast array of chemical, biological and radiological agents available for possible use in an intentional attack (USEPA, 2005c), deployment of contaminant-specific monitors that could specifically detect all of the chemical, biological, and radiological contaminants of concern would be impractical and cost prohibitive. In-depth reviews of contaminant-specific, emerging sensor technologies, and their roles in early warning systems, can be found elsewhere (ICF, 2005; States, et al., 2004). Instead of using contaminant-specific monitors throughout the distribution system, the design of the WS-CWS proposes using commercially available, rugged and cost-effective water quality monitors that are sensitive and responsive to a wide array of contaminants. Should a contaminant be introduced that results in a measurable change in water quality that is 'detected' as an anomaly by an event detection system (EDS), then site specific field testing and/or sampling with laboratory-based analyses would be performed to determine the specific contaminant that was introduced.

Online monitoring of water quality parameters, transmitting data in real-time to the utility's Supervisory Control and Data Acquisition (SCADA) system can serve the dual purpose of early detection of contamination and can save utility operators hours of sampling and testing time that would otherwise be required to collect even a fraction of the same data (Schlegal, 2004). Online monitors differ from handheld or stand alone monitors in that the data are continuously collected (at specified and frequent intervals), and can be transmitted in real or near-real time (results within minutes) to the SCADA system, or be processed by event detection algorithms prior to transmission to the SCADA. A partial listing of manufacturers of online water quality monitors is presented in **Table 2-1**. Hach Company (Loveland, Colorado) and YSI (Yellow Springs, Ohio) are two companies that make online monitors with data transmission capabilities. The Hach sensor stations can be equipped with Hach proprietary event detection system software that uses chemometrics (mathematical modeling of chemical data) to detect and characterize changes in water quality parameters (Kroll, 2005). Hach has compiled libraries of contaminant response profiles to achieve a level of contaminant specificity when the event detection system is triggered. Other companies, such as PureSense (Moffett Field, CA) and Source Sentinel LLC (Syracuse, New York), do not make water quality monitors but use commercially available monitors and sensors and their own algorithms to detect and transform the data from many monitors and sensors into a usable information stream that is transmitted to the SCADA system. Event detection systems (EDSs) are discussed in more detail in the Overview of Event Detection Systems for WaterSentinel (USEPA, 2005a).

Use of multiple water quality sensors provides corroborating information that may lead to more appropriate response decisions when a change in water quality is observed. For example, if one water quality parameter changes, an appropriate response may be to perform remote diagnostic testing on the sensor and review additional data and baseline trends to determine if the change is due to a harmless cause. However, if multiple water quality parameters change, a more appropriate response may be to dispatch site characterization teams to perform field tests and collect water samples from that area of the distribution system for further laboratory-based analyses. Automatic collection devices in the distribution system permit the capture of the water sample that triggers the response. Such a tiered approach for responding to anomalous water quality readings can be one means of managing the effort required to respond to triggers while still giving each trigger due consideration as a possible contamination threat (Hasan, et al., 2004; Alai, et al., 2005).

Table 2-1. Partial Listing of Manufacturers of Online Water Quality Monitors

#### Vendor

Dascore, Inc., (Jacksonville, FL) Hach Company, (Loveland, CO)

ManTech Associates, Inc., (Ontario, Canada)

Rosemount Analytical, Inc., (Irvine, CA)

YSI, Inc., (Yellow Springs, OH)

Applied Microsystems Ltd. (Sidney, BC, Canada)

Analytical Technology, Inc., (Collegeville, PA)

Isco, Inc., (Lincoln, NE)

Clarion Systems, Inc., (Indianapolis, IN)

GE/Sievers Ionics, Inc., (Boulder, CO)

Wallace and Tiernan Products (Vineland, NJ)

An important consideration in using online water quality monitors is the design and placement of sensor stations in the distribution system, and the capital and operating costs associated with installation and operation of an online water quality monitoring network. These topics are discussed in Sections 5.1 and 4.1, respectively, of *WaterSentinel System Architecture* (USEPA, 2005b). The remainder of this paper will summarize EPA and vendor studies that have been conducted to test the response of various water quality parameters to specific contaminants of concern, including some studies evaluating sensor performance during periods of extended operation. All the contaminant data presented in this paper are for concentrations well below the literature LD<sub>50</sub> values (*WaterSentinel Contaminant Fact Sheets*, USEPA, 2005d). Research is underway by the EPA's NHSRC to determine the health risks associated with consuming sub-acute concentrations of WS priority contaminants. As that information becomes available it will likely be used in the design of future studies.

**Table 2-2** presents a brief description of typical water quality parameters, how each is measured, and how utilities use the information to assess water quality and refine treatment, if necessary (Shaw Environmental Inc., 2004; American Society of Civil Engineers (ASCE), 2004). The parameters listed in Table 2-2 have been tested for their ability to respond to contaminants of concern; however, a smaller subset of parameters should form the core capability for the online water quality monitoring component of the WS-CWS. The parameters of pH, conductivity, total or free chlorine and TOC have proved to be the most sensitive and class specific responders to contaminants of concern.

**Table 2-2. Typical Water Quality Parameters** 

Parameter	Modes of Online Detection	How Information is Routinely Used
рН	Proton selective glass bulb electrode, proton selective metal oxide	pH is controlled for disinfection and corrosion control in distributions systems. Also, the formation of some disinfection byproduct (DBP) is pH dependant.
Conductivity	Annular ring electrode, nickel electrode, titanium or noble metal electrode	Ability of water to carry an electrical current. A strong indicator of the concentration of dissolved solids.
Free chlorine	Polarographic membrane, 3- electrode voltametric, colorimetric	Critical to demonstrate that disinfection requirements are met in the plant. In the
Total chlorine	Colorimetric	distribution system, chlorine residual monitoring is important to ensure that, 1) detectable residual levels are maintained at all points in the system as required by the SWTR; and 2) that maximum residual disinfectant levels are not exceeded as required by the Stage 1 D/DBP Rule. Also important for controlling biofilms, regrowth, nitrification, and other water quality problems.
ORP	Potentiometric, platinum or noble metal electrode	ORP values above 700 millivolts (mV) kill chlorine-sensitive organisms in drinking water. A groundwater incursion may lower ORP by increasing chlorine demand. Chlorination of drinking water produces an ORP background of ~700 mV.
тос	UV-persulfate digestion with near infrared detection or membrane conductometric detection of CO <sub>2</sub> .	Dissolved and particulate organic carbon. Concentrations in finished water can range from less than 1.0 mg/L to more than 10 mg/L. In any case, TOC concentrations are typically stable in distributed water from a single source. TOC is used as an indicator of DBP formation potential.
Dissolved oxygen (DO)	Membrane electrode, 3-electrode voltametric	Usually stable and near saturation for surface waters. A decrease may be an indication of chemical and biochemical activity in water.
Chloride	Ion selective electrode	Indicator of salinity.
Ammonia	Ion selective electrode	Naturally occurring form of nitrogen in the nitrogen cycle. May be added during treatment to form a combined chorine residual, which greatly reduces DBP formation. Excess ammonia can result in distribution system water quality problems such as nitrification.
Nitrate	Ion selective electrode	Essential nutrient for plants and animals. Nitrate is the most soluble form of nitrogen. For plants that have a combined distribution system residual, can be a result of nitrification. Drinkingwater maximum contaminant level (MCL) is 10 mg/L.
Turbidity	Nephelometric (light scattering) method	Indicator of suspended matter and microscopic organisms. Used as a process control tool in the plant to ensure that regulatory mandated treatment techniques for removal of pathogens are met. Limited application in distribution systems, but could be indicative of corrosion problems or other degradation in the quality of distributed water.

### Section 3.0: Literature Review

Prior to September 11, 2001, the vulnerability of the U.S. water supply was recognized but very little research had been done to improve monitoring of the distribution system (Clark and Deininger, 2000). Indeed, monitoring of the distribution system typically included a minimal set of water quality parameters for which data were collected at a low frequency. Models and physical testing show that water quality readings can change rapidly, and over a short timeframe in a backflow or injection attack (King and Kroll, 2005a). After September 11, 2001, research accelerated in an effort to determine if conventional water quality monitors could be deployed for extended periods of time and if the water quality parameters would respond to chemical and biological contamination. Research using online water quality monitors to detect contamination events has been performed sponsored primarily by manufacturers of water quality monitors (Table 2-1), EPA programs, the Edgewood Chemical Biological Center (ECBC) at Department of Defense, and the AwwaRF in collaboration with utilities and private companies.

Vendor and AwwaRF Studies - Hach Homeland Security Technologies (Hach HST), manufacturer of TOC analyzers and multi-parameter probes, is one of the few companies to have performed extensive testing of the ability of their equipment to respond to contaminants of interest to water security. They also have developed proprietary algorithms for event detection. An interesting add-on feature with the Hach HST system is a fully searchable library of contaminant signatures with the capability to add new contaminant profiles available to the end-user. To date, Hach HST has tested more than 80 chemicals (Kroll, 2004 and Kroll and King, 2005b) that include 12 of the chemicals on the WS contaminant list and 20 of the contaminants from the priority list (Table 3-1). This priority list was the starting point for the WS contaminant selection process through which 33 contaminants were identified for consideration in implementation in the initial WS pilot contaminants as described in *WaterSentinel System Architecture* (USEPA, 2005b). The results of the Hach tests have revealed that all the agents listed in Table 3-1. (subset of the 80 that have been tested) cause a distinct change in one or more of the measured parameters pH, turbidity, free chlorine, conductivity and TOC, and trigger Hach's Event Monitor<sup>TM</sup> Trigger System (EMTS) when tested at contaminant concentrations less than the LD<sub>50</sub> values in simulated distribution system studies (Kroll, 2004 and Kroll, 2005b).

Table 3-1. Select Contaminants that Trigger the Hach Event Monitor Trigger System

Aflatoxin B <sub>1</sub> (biotoxin)	Lead nitrate (toxic metal)
Aldicarb (insecticide)	Malathion (pesticide)
Arsenic trioxide (toxic metal)	Mercuric chloride (toxic metal)
Carbofuran (insecticide)	Methanol (industrial solvent)
Colchcine (anti-inflammatory)	Methomyl (agricultural insecticide)
Dicamba (pesticide)	Nicotine (insecticide, used free base)
Dichlorvos (insecticide)	Oxamyl (pesticide)
Diesel	Paraquat (herbicide)
E. coli (bacterial agent)	Phorate (insecticide)
Ethoprophos (nerve agent surrogate)	Sodium cyanide (toxic agent)
Ferricyanide (cyanide surrogate)	Sodium fluoroacetate (rodenticide)
Gasoline (hydrocarbon)	Strychnine nitrate (pesticide)
Glyphosate (herbicide)	Thallium (metal)

To address chemical warfare agents, Hach HST has entered into a Cooperative Research and Development Agreement (CRADA) with ECBC and the U.S. Army Corps of Engineers to assess which water quality parameters change in response to Sarin, Soman, VX, Ricin and Anthrax (Kroll, 2005b). ECBC is one of the few facilities in the country where testing that involves chemical or biological warfare agents can be conducted.

There are two AwwaRF studies in progress that will utilize online water quality monitors in a drinking water distribution system. In one of those studies, the contaminants sodium fluoroacetate, aldicarb, sodium arsenate and sodium cyanide have been tested, and detected, by online water quality monitors when injected into a recirculating pipe loop (Cook, et al., 2005). In the second AwwaRF study, event detection algorithms are anticipated to be tested using the data from online water quality monitors placed in the distribution systems of four different cities (AwwaRF, 2005).

**EPA Studies -** The EPA's **Technology Testing and Evaluation Program** (TTEP) provides unbiased third-party evaluation of commercially available homeland security related technologies. TTEP rigorously tests technologies against a wide range of performance requirements and specifications and posts summary reports and verification statements on their website (NHSRC, 2004). In the TTEP, the EPA is free to test, evaluate and compare performance of different vendor products. To date three different multi-parameter water quality probes have been tested by Battelle under the auspices of TTEP (NHSRC, 2005). The multi-parameter probes that were tested in the TTEP are listed in **Table 3-2**.

Table 3-2. Multi-Parameter Probe Technologies Tested by TTEP

Company	Instrument Model	Water Quality Parameters
YSI, Inc. (Yellow Springs, OH)	6600 Extended	DO, conductivity, temperature, pH,
131, Inc. (Tellow Springs, OT)	Deployment System	turbidity and chlorophyll
General Oceanics, Inc. (Miami, FL)	Ocean Seven 316	DO, conductivity, temperature, pH and turbidity
AANDERAA Instruments, Inc. (Norway)	RCM Mk II with Optode 3830	DO, temperature and turbidity

The three technologies were evaluated in field studies with respect to accuracy, relative bias, inter-unit reproducibility and precision in three waters over a period of extended deployment (2.5 months). In general, precision and accuracy was comparable for the common parameters (DO, conductivity, pH and temperature). Turbidity was found to be highly variable. In fact, turbidity is not a good primary indicator of contamination due to its high variability. The TTEP testing did not involve contaminant detection but does validate the precision, accuracy and bias of water quality probes in extended deployment environments such as would exist in the distribution system.

The Environmental Technology Verification (ETV) Program, established in 1995, uses voluntary vendor participation with stakeholder oversight to test and evaluate innovative technologies for use in environmental applications. In the ETV program, test plans are prepared with developers of new technologies and the tests conducted by an independent third party. After the results of those tests have been compiled and evaluated, verification reports and verification statements are posted on the EPA's ETV website (ETV, 2005). In 2005, five different multi-parameter online water quality probes were tested to independently verify the capability of the water quality monitors to respond and detect contamination events when deployed in a simulated distribution system. The reports of these studies are available in draft form but should appear on the EPA's ETV website when the reports are finalized (USEPA, 2005e,f,g,h,i). Two identical monitors from each vendor were tested before and after extended deployment for 52 days in a recirculating pipe loop distribution system simulator (DSS). The vendors, models and parameters tested are listed in Table 3-3.

Table 3-3. Multi-Parameter Probes Tested by the ETV Program for Contaminant Detection

Manufacturer	Model	Water Quality Parameters
Hach Company, (Loveland, CO)	Water Distribution Monitoring Panel (WDMP)	total chlorine, turbidity, temperature, conductivity, pH, and TOC
Rosemount Analytical, Inc. (Irvine, CA)	WQS Unit	free chlorine, turbidity, temperature, conductivity, and ORP
Clarion Systems, Inc. (Indianapolis, IN)	Sentinal 500	free chlorine, temperature, conductivity, pH, and ORP
Analytical Technology, Inc. (Collegeville, PA)	Q45WQ	free chlorine, turbidity, temperature, conductivity, pH, and ORP
ManTech Associates, Inc. (Ontario, Canada)	TitraSip™ SA	total chlorine, turbidity, temperature, conductivity, pH, and total alkalinity

The ETV tests were conducted by dissolving contaminant (nicotine, arsenic trioxide, aldicarb, and E. coli in growth medium) in five gallons of dechlorinated water and injecting the five gallons into a recirculating pipe loop of Cincinnati tap water. Tests were performed at the EPA's Test and Evaluation (T&E) Facility in Cincinnati, Ohio. The selected contaminants are on the WS contaminant list and represent organic, inorganic and biological contaminants. The final concentration of contaminant recirculating in the loop was 10 mg/L, well below the LD<sub>50</sub> values for these contaminants. To increase the solubility of the arsenic trioxide, the pH of the water used to dissolve the arsenic trioxide was adjusted to pH 12. Prior to, and at 3, 15 and 30 minutes after contaminant injection, water samples were collected and the water quality parameters were measured off-line using reference methods. A result was considered confirmed if a test probe detected a change in water quality as a result of contaminant injection that was also detected by the reference method. All contaminant injections were performed in duplicate. After 52 days of deployment in the recirculating pipe loop, additional experiments were performed to evaluate the ability of the probes to detect contaminant injections of aldicarb and E. coli in growth medium (E. coli represents the class of chlorine-sensitive contaminants on the WS contaminant list). A summary of the results from all five ETV tests are presented in **Table 3-4**. Table 3-4 indicates only if a change was detected; however, the directional change in water quality parameter can be found in the full ETV reports.

Table 3-4. Summary of Five ETV Studies Using Multi-Parameter Probes to Detect Contamination

Contaminant	Company	Total Cl <sub>2</sub>	Free Cl <sub>2</sub>	Turbidity	Temperature	Conductivity	рН	тос	ORP
	Hach	•		•				•	
	Rosemount		•						•
Nicotine	Clarion		•						•
	ATI		•	•					•
	ManTech	•		•					
	Hach	•		•		•	•		
Arsenic	Rosemount		•			•	•		•
Trioxide	Clarion		•			•	•		•
	ATI		•	•		•	•		•
	ManTech	•		•		•	•		
	Hach	•		•				•	
	Rosemount		•						•
Aldicarb	Clarion		•						•
	ATI		•	•					•
	ManTech	•		•					

Contaminant	Company	Total Cl <sub>2</sub>	Free Cl <sub>2</sub>	Turbidity	Temperature	Conductivity	рН	тос	ORP
	After Extended Deployment								
	Hach	•		•		•	•	•	
	Rosemount		•						•
E Coli	Clarion		•			•	•		•
	ATI		•	•			•		•
	ManTech	•		•		•	•		
	Hach	•		•			•	•	
Aldicarb	Rosemount		•						•
	Clarion		•				•		•
	ATI		•	•			•		•
	ManTech	•		•			•		

• = detectable change

-- = no observed change

Gray cells = parameter not monitored

Regardless of probe manufacturer or contaminant type, changes in total or free chlorine were able to detect the presence of contaminants, prior to, and after extended deployment. Since contaminant reaction with chlorine is an oxidation/reduction reaction, ORP tracked changes in free or total chlorine very well. The data would indicate that contaminant injections were detected by turbidity; however, there is less certainty with these data due to the large variability observed by the reference method. It is hypothesized that the turbidity changes may have been due to small air bubbles that could have been introduced during contaminant injection. Turbidity is thus not a good primary indicator of contaminant injection. Conductivity and pH changed with arsenic trioxide injection perhaps due to the pH adjustment required to get the arsenic trioxide in solution. Aldicarb and nicotine did not cause any changes in pH or conductivity in the pre-extended deployment experiments. In the extended deployment experiments, however, aldicarb appeared to elicit a change in pH in 4 of the 5 probes tested. The reference methods corroborated the changes detected by the probes yet it is not known why the pH did not change in previous experiments with aldicarb. TOC was able to detect the organic compounds aldicarb and nicotine, whereas, arsenic trioxide did not elicit a change in TOC.

Numerous studies involving contaminant detection using water quality monitors have been performed at the EPA's T&E Center in Cincinnati, Ohio (USEPA, 2005j); Water Information Sharing and Analysis Center (WaterISAC), 2005; Hall, et al., 2005). The specific program in which the research has been performed is the Water Assessment Technology Evaluation Research and Security (WATERS) Center. Within the WATERS Center there are multiple DSSs used to evaluate and understand the dynamics that influence water quality within the distribution system infrastructures typical in the U.S. Previously unpublished research performed by the EPA at the WATERS Center is presented as a Case Study below. Similar to the findings of other studies, total and free chlorine were the most sensitive water quality parameters, showing significant changes from baseline for the majority of chemicals and biological media tested. Changes in TOC were significant for organic contaminants and biological media. Conductivity was found to be a highly stable water quality parameter with response to contaminants tending to be detectable, but low. Turbidity response appeared to be sensitive but was highly erratic. The summary data presented in the Case Study show the percent changes in water quality parameters. The significance associated with that percent change is an estimation based upon the variability of the parameter typically observed in daily operations at the T&E Facility.

# **Section 4.0: Case Study**

Commercially available continuous, online sensors designed to monitor pH, free chlorine, total chlorine, ORP, DO, conductivity, turbidity, TOC, chloride, ammonia, and nitrate were tested in a pilot-scale system at the WATERS Center within the EPA's T&E Facility, Cincinnati, Ohio.

#### 4.1 Experimental

Two modes of distribution pipe simulation were tested: a recirculating pipe-loop and a single-pass pipe. **Table 4-1** contains typical values, with daily variation, for water quality parameters of Cincinnati tap water that was used at the T&E facility. Daily variation was approximately 10% for a volatile parameter like free chlorine and less than 2% for stable parameters like TOC and conductivity. Over a long time period (i.e. one year) values can vary by 20% or more, but this is due to seasonal and operational changes.

Table 4-1. Typical Water Quality Parameter Values with Daily Variability

Water Quality Parameter*	Typical Value
Total Chlorine	1.1 <u>+</u> 0.1 mg/L
Free Chlorine	1.0 <u>+</u> 0.1 mg/L
Total Organic Carbon	0.6 <u>+</u> 0.01 mg/L
Oxidation Reduction Potential	650 <u>+</u> 20 mV
Conductivity	375 <u>+</u> 5 μS/cm
pН	8.5 <u>+</u> 0.1
DO	7.0 <u>+</u> 0.1 mg/L
Nitrate-Nitrogen	4.0 <u>+</u> 0.1 mg/L
Chloride	30 <u>+</u> 2 mg/L
Turbidity	0.5 <u>+</u> 0.1 NTU

<sup>\*</sup>Data collected once every minute, except TOC which was collected once every 4 - 8 minutes.

In the recirculating pipe-loop mode, water continuously circulated through a 150-gallon capacity pipe-loop (75 feet by 6-inch diameter unlined, cast-iron) and a 100 gallon recirculation tank. A separate 30-gallon feed tank added Cincinnati Water Works chlorinated tap water to the loop at a rate (0.16 gallons per minute (gpm)) such that the entire volume of water in the loop and recirculation tank was replaced every 24 hours. Water quality monitors for total chlorine, free chlorine, ORP, conductivity, TOC, chloride, nitrate and ammonia were placed 70 feet downstream from the point of contaminant injection into the pipe-loop. Under these conditions, contaminants reached the monitors approximately 75 seconds after injection. The water quality parameters' response profiles reflected this design, with responses to the contaminants persisting until dilution, or chemical degradation via hydrolysis or reaction with free chlorine, resulted in a gradual return to baseline. The following contaminants were evaluated in the recirculating pipe-loop mode: potassium ferricyanide, a malathion insecticidal formulation (the insecticide Real Kill<sup>TM</sup>), a glyphosate herbicidal formulation (the weed killer Round-Up<sup>TM</sup>), and a secondary wastewater effluent. **Table 4-2** lists the online monitors tested in the recirculating pipe-loop mode.

Table 4-2. Online Monitors Tested in Recirculating Pipe-loop Mode

Single Parameter Probes	Water Quality Parameters			
ATI A15	Free chlorine			
Hach, CI-17*	Free and total chlorine			
Hach, 1720 D *	Turbidity			
GLI Model PHD*	рН			
GLI Model 3422*	Conductivity			
Hach Astro TOC UV Process Analyzer	TOC			
Multi-Parameter Probes	Water Quality Parameters			
Dascore, Six-Sense Sonde	Conductivity, DO, ORP, pH, temp, free chlorine			
YSI, 6600 Sonde	Conductivity, DO, ORP, pH, temp, ammonia- nitrogen, chloride, nitrate-nitrogen, turbidity			
Hach, Hydrolab Data Sonde 4a	Conductivity, DO, ORP, pH, temp, ammonia- nitrogen, chloride, nitrate-nitrogen, turbidity			

<sup>\*</sup>These sensors were contained within the same panel (Aguatrend).

In single-pass pipe mode, water flowed through a plastic-lined ductile iron pipe that was1,200 feet long by 3 inches in diameter in a single pass at 20 gpm (no recirculation). The same water quality monitors used in the recirculating pipe-loop experiments were used in the single-pass pipe experiments (**Table 4-3**). The specific water quality parameters tested were free chlorine, total chlorine, chloride, conductivity, DO, ORP, pH and turbidity. The monitors were placed at 80 and 1,100 feet downstream from the point of contaminant injection into the single-pass pipe. The water quality parameters' response profiles reflected a single-pass design, with responses to the contaminants persisting for approximately 20 minutes, with a rapid return to baseline. The following contaminants were evaluated in the single-pass pipe mode: aldicarb, glyphosate (not a store-bought formulation), colchicine (an anti-inflammatory), dicamba (an herbicide), dimethyl sulfoxide (DMSO) (solvent for chemical), lead nitrate, mercuric chloride, nicotine, potassium ferricyanide, sodium thiosulfate (reducing agent), sucrose (found in most biological growth media) and various growth media for biological contaminants.

#### 4.2 Results

Recirculating Pipe-Loop Mode – Event detection algorithms were not used in these experiments. Without event detection software, it was necessary to estimate the significance of changes in water quality parameters. This was done by establishing a stable baseline in parameter response prior to injection of the contaminant, and measuring the percent change of that response from baseline when the contaminant was injected. Knowledge of the within day variability (Table 4-1) was used to estimate the significance change in a parameter response from baseline. Table 4-3 shows the percent changes from baseline values for a small set of contaminants. Cells in the table (and in subsequent tables) that are highlighted in green indicate that the change in response was less than 10%. This was deemed to be a marginally significant change from baseline response. Cells in the table that are highlighted in yellow indicate that the change in response was between 10% and 50% which was deemed to be a significant change from baseline. Cells that are highlighted in red indicate a change greater than 50% which was deemed to be highly significant.

Table 4-3. Online Monitor Responses to Select Contaminants in Recirculating Pipe-Loop Mode\*\*

			Percent Change in Response From Baseline							
Contaminant	Amount Added	Total Cl <sub>2</sub>	Free Cl <sub>2</sub>	ORP	тос	CI	NO <sub>3</sub>	NH <sub>3</sub>		
Potassium	2 g*	10-50%	10-50%	<10%	10-50%	>50%	>50%	10-50%		
Ferricyanide	15 g*	>50%	>50%	10-50%	>50%	>50%	>50%	>50%		
Malathion	0.04 g*	<10%	<10%	<10%	10-50%	<10%	<10%	<10%		
Formulation	1.0 g*	10-50%	10-50%	<10%	10-50%	<10%	<10%	<10%		
Glyphosate Formulation	1.0 g*	>50%	>50%	10-50%	10-50%	<10%	<10%	<10%		
Secondary Wastewater Effluent	2 gallons	>50%	10-50%	10-50%	<10%	10-50%	10-50%	10-50%		

Green = marginally significant, Yellow = significant, Red = highly significant

No one sensor responded to all contaminants, however, total and free chlorine, ORP and TOC yielded the most significant responses to the widest variety of contaminants and concentrations tested. At the concentrations used in these experiments, the change in conductivity from baseline was less than 10%. Understanding the mode of detection for these online monitors is essential since each has interferences, fouling and maintenance issues that should be considered when interpreting the data (USEPA, 2005j). For example, the nitrate and chloride ion selective electrodes are likely responding to interference from the potassium ion in the potassium ferricyanide and not to nitrate or chloride ions.

Regardless of manufacturer, the online monitors were able to detect the presence of contaminants in the recirculating pipe-loop experiments with total and free chlorine, ORP, and TOC exhibiting the most significant changes with contaminant introduction. Further studies are planned to determine the threshold responses of the online monitors relative to toxicity and nuisance levels for real and surrogate contaminants.

**Single-Pass Pipe Mode** – **Table 4-4** shows the percent change from baseline for a subset of all the contaminants tested at three different concentrations in the single-pass pipe.

<sup>\*\*</sup>Monitor output recorded after 15 minutes of contaminant recirculation.

<sup>\*</sup>g = grams. Final concentrations of contaminants in circulation were 0.04 - 15 mg/L.

Table 4-4. Online Monitor Responses to Select Contaminants in Single-Pass Pipe Mode at 80 Feet from the Point of Injection

		Percent Change in Response From Baseline						
Contaminant	Initial Conc. In Pipe (mg/L)	Total Cl <sub>2</sub>		Free Cl <sub>2</sub>		ORP	CI	DO
Aldicarb	0.2	<10%		<10%		<10%	<10%	<10%
	1.1	10-50%		10-50%		<10%	<10%	<10%
	2.2	>50%		>50%		10-50%	<10%	<10%
Glyphosate	0.4	10-50%		10-50%		<10%	<10%	<10%
	1.5	10-50%		>50%		10-50%	<10%	<10%
	3.0	>50%		>50%		10-50%	<10%	<10%
DMSO	0.6	<10%		<10%		<10%	<10%	<10%
	2.0	<10%		10-50%		<10%	<10%	<10%
	4.0	10-50%		10-50%		<10%	<10%	<10%
Nicotine	0.4	<10%	<10%*	<10%	10-50%	<10%	<10%	<10%
	1.9	10-50%	10-50%*	10-50%	>50%	<10%	<10%	<10%
	3.8	10-50%	10-50%*	10-50%	>50%	10-50%	<10%	<10%
Potassium Ferricyanide	0.6	<10%		<10%		<10%	<10%	<10%
	1.6	10-50%		<10%		<10%	10-50%	<10%
	3.2	10-50%		<10%		<10%	10-50%	<10%
Sodium Thiosulfate	0.2	10-50%		10-50%		<10%	<10%	<10%
	1.3	>50%		>50%		10-50%	<10%	<10%
	2.6	>50%		>50%		10-50%	<10%	<10%

Green = marginally significant, Yellow = significant, Red = highly significant

Ion selective electrodes, like chloride, did not respond to contaminants as strongly in the single pass loop as in the recirculating loop. In general, recirculation experiments allow time for chemical reactions to approach equilibrium. Thus, while the single pass experiments more accurately simulate the actual hydraulics of a distribution system, the recirculation experiments better simulate reactions that proceed at a slower rate. The single-pass pipe mode represents the worst case scenario for injection of a contaminant: the sensors detect a contaminant close to the point of injection where reactions that may trigger the sensors have not fully developed.

Turbidity data are not presented in Table 4-4 since they were erratic and not a good primary predictor of contamination. Conductivity and pH were monitored and did not exhibit a change in baseline response greater than 5% for any contaminant or concentration tested so are not presented in Table 4-4. This does not mean that the changes were insignificant, especially for a very stable parameter such as conductivity which only varies approximately 2% from baseline over a day. Colchicine, dicamba, lead nitrate, mercuric chloride and sucrose were tested from 0.4-4.0 mg/L and elicited changes in baseline responses less than 10% for all contaminants and tested concentrations for all parameters. These data are not presented in the table for the sake of brevity.

**Figure 4-1** shows the change in free chlorine in response to injection of nicotine (3.8 mg/L) at two points in the single-pass pipe; 80 and 1,100 feet from injection. The experiments were performed in duplicate. It is apparent that the drop in free chlorine was greater at 1,100 feet than at 80 feet. This is due to the slower reaction kinetics of chlorine with nicotine.

<sup>\*</sup>Percent change at 1,100 feet. Nicotine showed a greater change in total and free chlorine at 1,100 feet from the point of injection due to slow reaction kinetics.

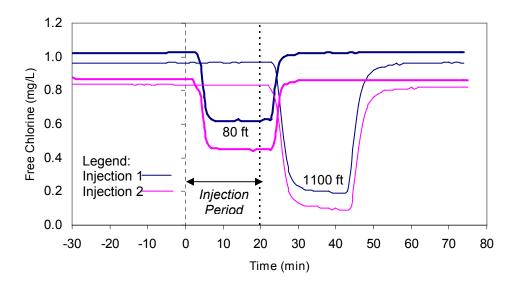


Figure 4-1. Change in Free Chlorine to Nicotine at 80 and 1,100 ft from Injection

**Figure 4-2** shows the response of free chlorine as a function of nicotine concentration. The figure shows the linear relationship between free chlorine loss and nicotine concentration and also shows that the rate of loss of free chlorine was greater at 1,100 feet than at 80 feet. This indicates that the reaction between chlorine and nicotine had not reached equilibrium in the very short travel times evaluated in these single pass experiments.

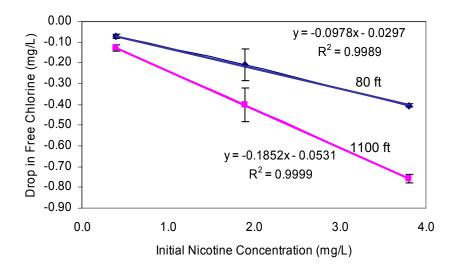


Figure 4-2. Free Chlorine Response with Increasing Initial Nicotine Concentration

The  $LD_{50}$  for nicotine is 50 mg/kg (oral, rat). This would correspond to a concentration in drinking water of 1,750 mg/L for a 70 kg person consuming 2 liters of water per day. The concentrations of nicotine tested in these experiments were between 0.4 and 3.8 mg/L, well below the drinking water equivalent

level of 1,750 mg/L. Similar sensitivity testing for other contaminants based on their toxicity should be performed in future research.

In separate experiments, common biological growth media at various concentrations were injected into the single-pass pipe and water quality parameters of total chlorine, free chlorine, chloride, conductivity, DO, ORP, pH, and turbidity were measured at 80 and 1,100 feet from the point of injection. Growth media were tested because in an intentional contamination event, a biological agent would most likely be injected into a pipe in growth media. **Table 4-5** contains only the results from measurements taken at 1,100 feet, because, similar to nicotine, more significant results were observed at 1,100 feet versus 80 feet from the point of injection due to reaction kinetics.

Table 4-5. Online Monitor Responses to Biological Growth Media in Single-Pass Pipe Mode at 1,100 Feet from the Point of Injection

Biological	Initial Conc.	Percent Change in Response From Baseline						
Growth Media	In Pipe (mg/L)*	Total Cl₂	Free Cl₂	ORP	CI	DO		
Nutrient Broth	0.12	<10%	<10%	<10%	<10%	<10%		
	0.48	<10%	<10%	<10%	<10%	<10%		
	0.96	<10%	10-50%	<10%	<10%	<10%		
Trypticase Soy Broth	0.12	<10%	<10%	<10%	<10%	<10%		
	0.48	10-50%	10-50%	<10%	<10%	<10%		
	0.96	10-50%	10-50%	<10%	<10%	<10%		
Terrific Broth	0.12	<10%	<10%	<10%	<10%	<10%		
	0.47	<10%	10-50%	<10%	<10%	<10%		
	0.97	<10%	10-50%	<10%	<10%	<10%		
<i>E. coli</i> in Terrific Broth	0.12	10-50%	10-50%	<10%	<10%	<10%		
	0.47	10-50%	>50%	<10%	<10%	<10%		
	0.97	10-50%	>50%	<10%	10-50%	<10%		

Green = marginally significant, Yellow = significant, Red = highly significant

It is apparent from Table 4-5 that free and total chlorine are the water quality parameters most sensitive to biological contaminant incursion.

Total and free chlorine were the most sensitive water quality parameters, showing significant changes from baseline in both the recirculating and single-pass pipe experiments. Changes in TOC were significant for organic contaminants in recirculating pipe-loop mode. Conductivity and pH did not display changes in response greater than 5% for the contaminants and concentrations tested; however, the concentrations used in most experiments were well below lethal concentrations. Again, turbidity was erratic and not a good primary predictor of contamination. In the single-pass pipe mode, ORP showed marginally significant to significant change in baseline response.

To distinguish the normal variability of water quality parameters from real anomalies that could indicate contamination, it is necessary to use an EDS (USEPA, 2005a). It should be noted that results from the EPA's T&E experiments were reported as an observed percent change from an estimated baseline and that the use of an event detection system may have resulted in lesser or greater sensitivity to these contaminants. Future work should utilize event detection software and test a wider variety of chemical and biological contaminants at concentrations of interest. Online monitoring of radiological contamination has unique considerations that should also be addressed in future research.

<sup>\*</sup>Concentration of growth media.

# **Section 5.0: Summary and Conclusions**

In summary, the work done by manufacturers of online water quality monitors and EPA demonstrate that online water quality monitors are sensitive and responsive to contaminant incursion (Kroll, 2005a; Shaw Environmental Inc., 2004; Kroll and King, 2005a; Cook, et al., 2005; USEPA, 2005j; WaterISAC, 2005; Hall, et al., 2005; Kroll and King 2005c). The water quality parameters most sensitive to contaminants of concern to water security are free or total chlorine, total organic carbon, conductivity and pH. Oxidation/reduction potential corroborates the chlorine sensor results. Other parameters such as chloride, nitrate and ammonia have been observed to change in the presence of contaminants but mostly due to interferences of concomitant ions. Turbidity, which can be highly variable, is not a good primary indicator of contamination

The WS contaminant selection process identified 33 contaminants for consideration in implementation of the WS pilot. Based on the means by which contaminants could be detected in the proposed WS contaminant warning system, EPA classified contaminants into 12 categories (*WaterSentinel Contaminant Selection, 2005k*). Online water quality monitoring data from distribution system simulation studies were used to inform the process of contaminant classification. Contaminants that impact two or more water quality parameters have a high potential for detection by online monitoring. If the presence of the contaminant only has an impact on a single water quality parameter, online monitoring provides a moderate potential for detection.

Online water quality monitoring is just one component in the WS System Architecture (USEPA, 2005b). The purpose of online water quality monitoring in the WS design is to serve as a means for a utility to detect potential contamination and initiate an appropriate response. The subsequent role of field and laboratory testing is to determine if a contamination event occurred and, if so, what contaminant was introduced.

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# **Appendix A: Acronym List**

ASCE American Society of Civil Engineers

AwwaRF American Water Works Association Research Foundation
CRADA Cooperative Research and Development Agreement

CWS contamination warning system

DBP disinfection byproduct
DMSO dimethyl sulfoxide
DO dissolved oxygen

DSS distribution system simulator

ECBC Edgewood Chemical Biological Center

EDS event detection system

EMTS Event Monitor<sup>TM</sup> Trigger System

EPA U.S. Environmental Protection Agency
ETV Environmental Technology Verification
Hach HST Hach Homeland Security Technologies

gpm gallons per minute

MCL maximum contaminant level

mV Millivolts

NHSRC National Homeland Security Research Center

ORD Office of Research and Development

ORP oxidation/reduction potential SCADA Supervisory Control and Data SWTR Surface Water Treatment Rule

T&E Test and Evaluation TOC total organic carbon

TTEP Technology Testing and Evaluation Program

WATERS Water Assessment Technology Evaluation Research and Security

WaterISAC Water Information Sharing and Analysis Center

WDMP Water Distribution Monitoring Panel

WS WaterSentinel

WS-CWS WaterSentinel contamination warning system

WSD Water Security Division